

ICS 77.100.40

## **DRAFT EAST AFRICAN STANDARD**

SIIC

# Synthetic and combined (soap and synthetic) liquid hand wash --Revi **Specification**

2.40'

### **EAST AFRICAN COMMUNITY**

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#### Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS) and other deliverables. The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards.

East African Standards and other deliverables are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 074, Surface active agents

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

This second edition cancels and replaces the first edition (EAS 812-1:2015), which has been technically revised.

#### Introduction

Liquid hand wash has become very common nowadays as liquid soaps are easy to use, even for children.

They come in different types and most types of liquid hand wash have moisturizers and anti-oxidants in their formulations, such as Vitamin E or Aloe, that help to protect the skin. Permitted fragrances and dyes are among some of the other ingredients used in their formulations.

Liquid hand wash on the market are either soap based, combined (soap and synthetic) or pure synthetic with most products being either synthetic or combined (soap and synthetic).

Due to the scarcity of fats and oils, technology is moving towards the synthetic type of hand wash as opposed to soap based.

uality . For public the second This necessitated the development of this standard to guide the quality and safety of synthetic and combined

# Synthetic and combined (soap and synthetic) liquid hand wash – Specification

#### 1 Scope

This Draft East African Standard specifies the requirements, sampling and test methods for synthetic and combined (soap and synthetic) hand wash.

This standard does not apply to only soap-based hand wash.

#### 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ASTM D1569, Standard test method for detergent alkylate

EAS 217-1-1, Methods for the microbiological examination of foods — Part 1-1: General procedures and techniques

EAS 377-1, Cosmetics and cosmetics products — Part 1: List of substances prohibited in cosmetic products

EAS 377-2, Cosmetics and cosmetics products — Part 2: List of substances which cosmetic products must not contain except subject to restrictions laid down

EAS 377-3, Cosmetics and cosmetics products — Part 3: List of colourants allowed in cosmetic products

EAS 377-4, Cosmetics and cosmetics products - Part 4: List of preservatives allowed in cosmetic products

EAS 377-5, Cosmetics and cosmetics products — Part 5: Use of UV filters in cosmetic products

EAS 794, Determination of the microbial inhibition of cosmetic soap bars and liquid hand and body washes — Test method

ISO 4314, Surface active agents — Determination of free alkalinity or free acidity — Titrimetric method

ISO 862, Surface active agents - Vocabulary

ISO 4316, Surface active agents — Determination of pH of aqueous solutions — Potentiometric method

#### 3 Terms and definitions

For the purposes of this standard terms and definitions given in ISO 862 shall apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at http://www.iso.org/obp

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#### 4 Requirements

#### 4.1 General requirements

**4.1.1** All ingredients used in the liquid hand wash shall comply with the requirements of all parts of EAS 377.

**4.1.2** If humectants, for example glycerine or propylene glycol, are part of the formulation, the total amount should not be less than 1 % m/m.

4.1.3 The liquid hand wash shall not have any objectionable odour.

**4.1.4** The liquid hand wash shall not be irritating to the normal skin, and shall not contain ingredients in quantities that are toxic to human beings.

**4.1.5** If the liquid hand wash contains herbal extracts, the total herbal extracts (for example neem or Aloe Vera) amount shall not be less than 0.01 % m/m of the 100 % (pure) active ingredient.

#### 4.2 Specific requirements

The liquid hand wash shall also comply with the specific requirements given in Table 1 when tested in accordance with the corresponding test method.

SI No	Characteristic	Re	Test method		
		Synthetic	Combined (synthetic and Soap)		
i)	Total active content, min, % m/m, min	5	10	Annex B	
ii)	pH at 20°C, neat, range	3.5 <sup>a)</sup> – 8	3.5 <sup>a)</sup> – 8	ISO 4316	
iii)	Specific gravity at 20°C	0.85 - 1.40	0.85 - 1.40	ASTM D1569	
iv)	Free fatty acid content (as oleic acid), % m/m, max.	_	1	ISO 4314	
v)	Lather volume, ml, min.,	110	110	Annex A	
vi)	Total plate count, cfu/g. max	100	300	EAS 217-1-1	
~	Yeast & Mold, cfu/g max	100	100		
vii)	Antibacterial activity <sup>b)</sup>	To pass the test		EAS 794.	
	ak acids should be used such as lactic acid, citric acid liquid hand wash containing antibacterial agents claim				

#### Table 1 — Specific requirements for liquid hand wash

#### 5 Packaging

5.1 The liquid hand wash shall be packaged in suitable well-closed containers.

**5.2** The containers/packages (including the closures) shall not impart any contamination to the liquid hand wash and shall be strong enough to protect the content adequately during normal handling, transportation and storage.

**5.3** Only containers/packages of the same size and bearing the same batch identification shall be packed together in a bulk container/package.

#### 6 Labelling

Each container and bulk package shall be securely closed, legibly and indelibly labelled either in English, Kiswahili or French or combination or any other language as agreed between the manufacturer and supplier with the following information:

- a) name of the product as "liquid hand wash";
- b) indication of whether the product is synthetic or combined (soap and synthetic)
- c) manufacturer's name and physical address;

NOTE: The name, physical address of the distributor/supplier and trade mark may be added as required.

- d) batch or lot number;
- e) net content;
- f) list of ingredients used;
- g) country of origin;
- h) instructions for use
- i) date of manufacture; and
- j) best before date.
- k) indicate mode of disposal of containers/packages

#### 7 Sampling

Sampling shall be done in accordance with Annex C.

#### Annex A (normative) Test for lather volume

#### A.1 General

Strict attention shall be paid to all details of the procedure in order to ensure concordant results. Particular care should be taken to invert the cylinder exactly as described.

#### A.2 Outline of the method

A suspension of the material in standard hard water is taken in a graduated cylinder and given 12 inversions under prescribed conditions. The volume of the foam formed is observed after keeping the cylinder for 5 minutes.

#### A.3 Reagents

- A.3.1 Calcium chloride CaCl<sub>2</sub>.2H<sub>2</sub>O, AR
- A.3.2 Magnesium sulphate MgSO<sub>4</sub>.7H<sub>2</sub>O, AR
- A.3.3 Distilled water

#### A.4 Apparatus

**A.4.1** Graduated cylinder — Glass stoppered with graduation from 0 to 250 ml, with 2 ml divisions. Overall height about 35 cm and the height of the graduated portion about 20 cm.

#### A.4.2 100-ml glass beaker

A.4.3 Thermometer of range 0 - 110 °C

#### A.5 Preparation of standard hard water

Dissolve 0.220 g of calcium chloride dihydrate and 0.246 g of magnesium sulphate heptahydrate in distilled water. Dilute to 5 L with distilled water.

NOTE This standard hard water has a hardness of approximately 50 ppm calculated as calcium carbonate.

#### A.6 Procedure

**A.6.1** Weigh 1 g of the hand wash accurately in a 100-ml glass beaker. Add 10 ml of the standard hard water. Cover the beaker with a watch glass and allow to stand for 30 min. The operation is carried out to disperse the hand wash.

**A.6.2** Stir the contents of the beaker with a glass rod and transfer the slurry to a 250-ml graduated cylinder ensuring that not more than 2 ml foam is produced. Repeat the transfer of the residue left in the beaker with further portions of 20 ml of standard hard water ensuring that all the matter in the beaker is transferred to the cylinder.

**A.6.3** Adjust the contents in the cylinder to 100 ml by adding sufficient standard hard water. Bring the contents of the cylinder to 30 °C. Stir the contents of the cylinder with a glass rod or thermometer to ensure a uniform suspension.

**A.6.4** As soon as the temperature of the contents of the cylinder reach 30 °C, stopper the cylinder and give it 12 complete inversions, each inversion comprising movements in a vertical plane, upside down and vice versa. After the 12 inversions, let the cylinder stand for 5 min. Take the following readings as shown in Figure A.1:

- a) foam plus water (V<sub>1</sub> ml).
- b) water only (V<sub>2</sub> ml).

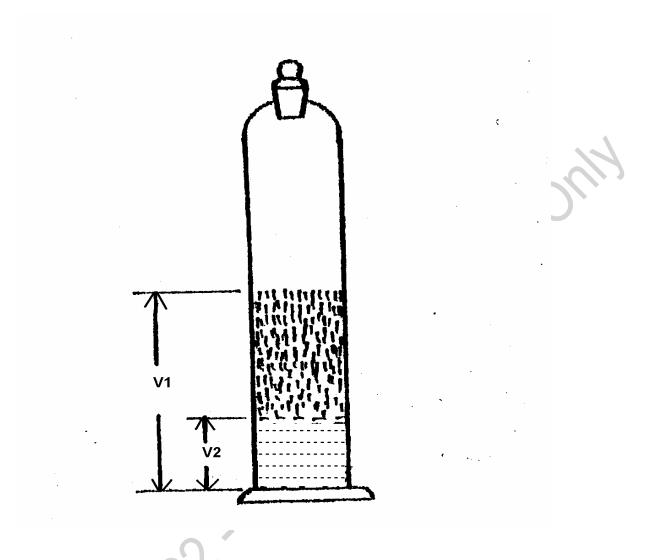


Figure A.1 — Measurement of foam

#### A.7 Calculation

Lather volume =  $V_1 - V_2$ 

where

 $V_1$  = Volume, in ml of foam + water;

 $V_2$  = Volume, in ml of water only.

#### Annex B (normative)

#### Determination of active detergent content

#### B.1 Outline of the method

When equivalent amounts of cationic and anionic detergents are present in a two-phase mixture of water and chloroform, methylene blue will colour the two phases to the same degree. Sodium alkyl benzene sulphonate and sodium lauryl sulphate or any other detergent can be titrated with a standard solution of cetyl trimethyl ammonium bromide.

#### **B.2** Reagents

Weigh  $1.5 \pm 0.001$  g of cetyl trimethyl ammonium bromide into a 250 ml beaker. Add 100 ml of distilled water and stir until dissolved. Transfer quantitatively to a 1 litre volumetric flask and make to volume. Mix thoroughly and standardize against solution B. (See B.2.1).

#### **B.2.1** Anionic solution (Solution B)

Weigh accurately such amount of standard alkyl sulphate of known combined SO<sub>3</sub> or active content so as to give exactly 0.320 g of combined SO<sub>3</sub> into a 250 ml beaker. Dissolve in 100 ml to 200 ml of warm water. Transfer quantitatively to 1-litre volumetric flask and make to volume with water at room temperature. Mix thoroughly. This is the primary standard against which solution A, is standardized. Solution B is 0.004 N.

#### **B.2.2 Methylene blue indicator**

Dissolve 0.1 g of methylene blue in 100 ml of water. Transfer 30 ml of this solution to a 1-L flask. Add 500 ml of water, 6.8 ml of concentrated sulphuric acid, 50 g of sodium phosphate ( $NaH_2PO_4H_2O$ ) and shake until solution is complete. Dilute to the mark.

#### **B.2.3 Chloroform**

Analytical reagent grade

#### **B.3** Procedure

**B.3.1** Weigh accurately a sample of sufficient size to give approximately 0.320 g of combined SO<sub>3</sub> into a 250 ml beaker. Sample size is crucial (see Note). Use 700 ml to 800 ml of warm water to transfer quantitatively to a 1-L volumetric flask. Warm on steam bath and shake gently until the sample is dissolved and solution is clear. Cool, dilute to the mark and mix thoroughly.

NOTE The titration value V should be as near as to 10 ml as possible, say between 8 ml and 12 ml but never outside 5 ml and 15 ml.

**B.3.2** Pipette 10.0 ml of the sample solution into a 100 ml glass stoppered cylinder (25 x 300 mm). Add 25.0  $\pm$  0.5 ml of methylene blue solution and 10  $\pm$  0.5 ml of chloroform (see Note). Titrate with solution A to the correct end point, shaking the cylinder carefully after such addition to avoid emulsion and maintaining temperature within prescribed limits of 20 °C - 30 °C by immersion in water bath, if necessary. As the end point is approached, the rate of transfer of colour increases and solution A shall be added dropwise with vigorous shaking after each addition. If the approximate titration is known, before shaking since this avoids emulsion formation.

Application of vacuum to the titration cylinder may help to break some emulsions, if formed. The end point is reached when both layers have same colour intensity. The end point is very sharp and 0.05 ml will cause a distinct change in colour distribution at or near the equivalence point.

NOTE The titration value V should be as near to 10 ml as possible, say between 8 ml and 12 ml but never outside 5 ml and 15 ml.

**B.3.3** Pipette 10.0 ml of the sample solution into a 100-ml glass stoppered cylinder (25 x 300 mm). Add 25.0 ml  $\pm$  0.5 ml of chloroform (see Note). Titrate with solution A to the correct end point, shaking the cylinder carefully after such addition to avoid emulsion and maintaining temperature within prescribed limits of 20 °C to 30 °C by immersion in water bath if necessary. As the end point solution A shall be added dropwise with vigorous shaking after each addition. If the approximate titration is known, 80 % of the required titrating solution should be added before shaking since this avoids emulsion formation.

Application of vacuum to the titration cylinder may help to break some emulsions, if formed. The end point is reached when both layers have same colour intensity. The end point is very sharp and 0.05 ml will cause a distinct change in colour distribution at or near equivalence point.

NOTE The volume of methylene blue solution and chloroform may be changed if found advantageous provided the same volumes are used in standardizing solutions A and B.

#### **B.3.4 Calculation**

**B.3.4.1** The percent combined SO<sub>3</sub> shall be expressed as follows:

% combined SO<sub>3</sub> =  $\frac{V \times N \times 8.0}{M}$ 

where,

- V volume, in millilitres, of solution A used in the titration;
- *N* normality of solution A; and
- *M* mass, in grams, of the sample in the aliquot.

**B.3.4.2** The percent active detergent content shall be expressed as follows:

Percent active detergent content = percent combined SO<sub>3</sub> x Mol. weight of active detergent.

NOTE The molecular weight of active detergent should be supplied by the manufacturer on request.

#### B.4 Alternative method for determination of active detergent content

(To be used only if the first method (Clause B.1) fails to work on the product).

#### **B.4.1 Field of application**

This method is applicable to the analysis of alkylbenzene sulphonates, alkyl sulphonate, sulphates and hydroxy-sulphates, alkylphenol and fatty alcohol ethoxysulphates and dialkyl sulphosuccinates and to the determination of active materials containing one hydrophilic group per molecule.

#### B.4.2 Principle

Determination of anionic-active matter in a medium consisting of an aqueous and chloroform phase, by volumetric titration with a standard cationic-active solution (benzethonium chloride), in the presence of an indicator which consists of a mixture of a cationic dye (dimidium bromide) and an anionic dye (acid blue 1).

#### **B.4.3 Reagents**

**B.4.3.1** The water used shall be of distilled quality.

**B.4.3.2** Chloroform, (sp. gravity = 1.48 g/m, distilling between 59.5°C and 61.5°C).

**B.4.3.3** Sulphuric acid, 2.5 M solution.

**B.4.3.4** Sulphuric acid, 0.5 M solution.

**B.2.3.5** Sodium hydroxide, 1.0 M standard volumetric solution.

**B.4.3.6** Sodium lauryl sulphate (sodium dodecyl sulphate) CH<sub>3</sub>(CH<sub>2</sub>)<sub>11</sub>OSO<sub>3</sub>Na 0.004 M standard volumetric solution.

Check the purity of the sodium lauryl sulphate and simultaneously prepare the standard solution.

**B.4.3.6.1** Determination of purity of sodium lauryl sulphate — Weigh to the nearest 1 mg, 5 g  $\pm$  0.2 g of the product into a 250-ml round bottom flask with ground glass neck. Add exactly 25 ml of the sulphuric acid solution (B.4.3.4) and reflux into a water condenser.

During the first 5 min - 10 min, the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask.

In order to avoid excessive foaming, instead of refluxing the solution may be left on a boiling water bath for 60 min.

After a further 10 min the solution clarifies and foaming ceases. Reflux for further 90 min. Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water.

Add a few drops of the phenolphthalein solution (B.4.3.8) and titrate the solution with the sodium hydroxide solution (B.4.3.5).

Carry out a blank test by titrating 25 ml of the sulphuric acid solution (B.4.3.4) with the sodium hydroxide solution (B.4.3.5).

The purity of the sodium lauryl sulphate, expressed as a percentage,

$$=\frac{28.84(V_{1}-V_{0})\ M_{0}}{M_{1}}$$

where,

 $V_0$  = volume, in millilitres, of sodium hydroxide solution used for the blank test;

 $V_1$  = volume, in millilitres, of sodium hydroxide solution used for the sample;

 $m_1 = \text{mass}$ , in grams, of the sodium lauryl sulphate to be checked; and

 $M_0$  = exact molarity of the sodium hydroxide solution.

**B.4.3.6.2** Weigh 0.004 M sodium lauryl sulphate standard volumetric solutions. Weigh, to the nearest 1 mg between 1.14 g and 1.16 g of sodium lauryl sulphate and dissolve in 200 ml of water. Transfer to a ground glass stoppered 1-L volumetric flask and dilute to the mark with water.

Calculate the molarity,  $M_1$ , of the solution by means of the solution by means of the formula:

$$M_1 = \frac{m_2 \times purity(\%)}{288.4 \times 100}$$

where,

 $m_2$  = mass in grams of sodium lauryl sulphate.

**B.4.3.7** Benzethonium chloride 0.004 M standard volumetric solution

Weigh, to the nearest 1 mg, between 1.75 g and 1.85 g benzethonium chloride and dissolve in water. Transfer to a ground glass-stoppered 1-L volumetric flask and dilute to the mark with water.

NOTE In order to prepare a 0.004 M solution, dry the benzethonium chloride at 105EC, weigh 1.792 g, to the nearest 1 mg, dissolve in water and dilute to 1 L.

**B.4.3.8** Phenolphthalein, ethanolic solution containing 10 g/L. Dissolve 1 g of phenolphthalein in 100 ml of 95 % (v/v) ethanol.

#### B.4.3.9 Mixed indicator

#### B.4.3.9.1 Stock solution

Weigh to the nearest 1 mg 0.5 g  $\pm$  0.005 g dimidium bromide into a 50-ml beaker, and 0.025 g  $\pm$  0.005 g of acid blue 1 into a second 50-ml beaker.

Add between 20 ml and 30 ml of hot 10 %. (v/v) ethanol to each beaker. Stir until dissolved and transfer the solutions to a 250-ml volumetric flask. Rinse the beakers into the volumetric flask with ethanol and dilute to the mark with 10 % (v/v) ethanol.

#### B.4.3.9.2 Mixed acid indicator solution

Take 20 ml of the stock solution prepared above, put it in a 500-ml volumetric flask. Add 200 ml of water, and 20 ml of 2.5 M sulphuric acid (B.4.3.3) mix and dilute to the mark with water. Store away from direct sunlight.

#### **B.4.4** Apparatus

Ordinary Laboratory apparatus, and

- a) bottles, 200-ml, glass stoppered, or measuring cylinders, flask stoppered.
- b) burettes, 25-ml and 50-ml.
- c) volumetric flask, 1-L capacity glass stoppered.
- d) pipette, 25-ml.

#### B.4.5 Procedure

#### B.4.5.1 **Standardization of benzethonium chloride solution**

By means of the pipette transfer 25 ml of the 0.004 M sodium lauryl sulphate solution to a bottle or measuring cylinder, add 10 ml of water, 15 ml of the chloroform and 10 ml of the mixed indicator solution.

Titrate with the 0.004 M benzethonium chloride solution. Stopper the bottle or measuring cylinder after each addition and shake well. The lower layer will be coloured pink. Continue the titration with repeated vigorous shaking. As the end point approaches, the emulsions formed during shaking tend to break easily continue the titration drop by drop. Shaking after each addition of titrant, until the end point is reached. This is at the moment when the pink colour is completely discharged from the chloroform layer, which becomes a faint greyish blue.

The molarity, M, of the benzethonium chloride solution is given by the formula:

$$M = \frac{M_1 \times 25}{V_2}$$

where,

- $M_1$  molarity of the sodium lauryl sulphate solution; and
- V<sub>2</sub> Volume, in mollolitres, of benzethonium chloride added.

#### B.4.5.2 Determination

Weigh to the nearest 1 mg a sample of 30 g; dissolve the test portion in water. Add a few drops of the phenolphthalein solution and neutralize to a faint pink colour with the sodium hydroxide solution or sulphuric acid solution as required.

Transfer to a 1-L volumetric flask and dilute to the mark with water. Mix thoroughly and, by means of the pipette transfer 25 ml of this solution to a bottle or measuring cylinder, add 10 ml of water, and add 10 ml of water, and 15 ml of chloroform. Titrate with the benzethonium chloride solution as described in B.4.5.1.

#### **B.4.6 Expression of results**

The content as a percentage by mass, of anionic-active matter

$$= \frac{V_3 \times M \times 1000 \times M_0 \times 100}{25 \times 1000 \times M_0}$$

 $=4V_3M$ 

The amount of active matter, expressed in milliequivalents per gram,

$$\frac{=40 \times V_3 \times M_1}{M_0}$$

where,

- Mo mass, in grams, of the test portion;
- M relative molar mass of anionic-active matter;
- $M_1$  molarity of the benzethonium chloride solution;
- $V_3$  volume, in millilitres, of benzethonium chloride solution used for the titration of a 25-ml aliquot of anionic-active matter solution.

#### Annex C (normative)

#### Sampling

#### C.1 Procedure

**C.1.1** In a single consignment, all packages (cartons) containing toilet soap cakes drawn from the same batch of production shall constitute a lot. For ascertaining the conformity of the lot to the requirements of this standard, tests shall be carried out on each lot separately. The number of packages to be selected for drawing the sample shall be in accordance with Table C.1.

Number of packages (cartons) in the lot	Number of packages (cartons) to be selected	Number of samples		
N	n			
4 to 15	3	3		
16 to 40	4	4		
41 to 65	5	2		
66 to 110	7	2		
111 and above	10	1		

Table C	.1 —	Scale	of s	samp	ling
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**C.1.2** The packages shall be selected at random, using tables of random numbers. If these are not available, the following procedure shall be applied:

Starting from any package, count all the packages in one order as 1, 2, 3.... *N*, selecting every  $k^{\text{th}}$  package, where *k* is the integral part of  $N \div n$ .

**C.1.3** From each package thus selected, draw at random an equal number of bottles so as to obtain a total volume of at least 2 L.

#### C.2 Preparation of test samples

#### C.2.1 Composite sample

Weigh each cake separately (including any material that may have adhered to the wrapper), and calculate the average mass. Cut each of the remaining cakes into eight parts by means of three cuts at right angles to each other through the middle. Grate finely the whole of two diagonally opposite eighths of each specimen. Mix the gratings and place in a clean, dry, airtight glass container.

#### C.2.2 Samples for testing

Immediately after preparation of composite sample (C.2.1), take at one time all test samples required for the tests in 4.2. Weigh out the test sample required for determination of free alkali or acid content, and use it immediately.

#### Bibliography

ent EAS 812-1:2013 Liquid hand wash — Specification — Part 1: Synthetic and combined (soap and synthetic) hand wash.